



MORRISON KNUDSEN CORPORATION

## VASQUEZ BLVD./I-70 REMEDIAL INVESTIGATION TECHNICAL MEMORANDUM

**TO:** Bonnie Lavelle **DATE:** July 10, 2000

**FROM:** Marta Green

**RE:** RAC Contract No. 68-W7-0039  
WA 004-RICO-089R

**SUBJECT:** DRAFT DATA QUALITY ASSESSMENT  
PHASE III SAMPLING PROGRAM

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### OVERVIEW

Chemical analysis of the Vasquez Boulevard/Interstate 70 (VB/I-70) site characterization samples was conducted under a comprehensive quality assurance program. The program includes requirements for the collection, preparation, and analysis of quality control samples, as specified in the Project Plan for the Vasquez Boulevard & I-70 Site, Phase III Field Investigation (ISSI, 08/04/99), Section 4.0 Quality Assurance Project Plan (QAPP), and related Standard Operating Procedures for sample collection, preparation and analysis.

An assessment of the data quality was performed throughout the program on a daily basis to verify compliance with the quality control criteria and to identify necessary corrective actions. An assessment of all Phase IIIA data, including residential surface soil, garden soil, garden vegetables and indoor dust, has been performed to verify that the data set is consistent with and meets the data quality objectives identified in the QAPP. The data quality assessment is presented in terms of the precision, accuracy, representativeness, comparability, and completeness of the data. The results document that the data are usable for their intended purpose of identifying average surface soil concentrations and supporting the Baseline Risk Assessment.

## SOIL SAMPLE DATA QUALITY

Soil samples were collected from residential yards, vegetable gardens, schools, and a park. All soil samples were prepared in the field laboratory by homogenizing the sample, drying a portion of the sample, sieving the sample through a #10 sieve, and then grinding a portion of the sieved, bulk fraction. The ground sample was analyzed at the field laboratory using a QuanX Energy Dispersive X-Ray Fluorescence Spectrometer (XRF). A percentage of samples were split and also submitted for off-site laboratory analysis.

Quality control sample results for soils analyzed by XRF are charted in Figures 1 through 17. Table 1 summarizes the number of soil field samples and each type of quality control sample.

### **Precision**

Precision measures the reproducibility of values under a given set of conditions. Precision was measured in Phase III soils through preparation and analysis of laboratory duplicates and blind split samples.

#### *Laboratory Duplicates*

Laboratory duplicates were prepared and analyzed at a frequency of one for every twenty field samples. Laboratory duplicates were identifiable to the analyst so that the duplicate and original field sample results could be reviewed immediately following analysis. The results of the laboratory duplicates are presented in Figures 1 through 4. Duplicates met the quality control criteria of less than 25% relative percent difference between the original sample and its duplicate, or less than one method detection limit (MDL) for samples with concentrations less than five times the MDL, in all but four samples for arsenic and two samples for lead. The results for samples associated with the preparation of these six duplicates exceeding the precision criteria were qualified as estimated. Overall correlation of original samples versus duplicates was very good.

#### *Blind Splits*

Blind split samples were prepared at the same frequency and in the same manner as laboratory duplicates, but were assigned a unique sample identification number and submitted blind to the analyst such that it could not be distinguished from other field samples. The results of the laboratory duplicates are presented in Figures 5 through 8. Blind splits met the quality control criteria of less than 25% relative percent difference between the original sample and its duplicate, or less than one MDL for samples with concentrations less than five times the MDL, in all but five samples for arsenic and three samples for lead. The results for samples associated with the preparation of these eight blind splits exceeding the precision criteria were qualified as estimated. Overall correlation of original samples versus blind splits was very good.

### **Accuracy**

Accuracy measures the bias from the true value in a measurement system. Analytical accuracy was evaluated in soils through determination of the arsenic and lead MDLs, instrument calibration using certified standard reference materials (SRM), and analysis of blind standards.

#### *Method Detection Limit Study*

The MDL is the lowest concentration of a substance that can be measured and reported with a 99% confidence that the analyte is present. An instrument- and matrix-specific MDL was determined in Phase III for arsenic and lead. MDL studies were conducted prior to XRF analysis of field samples, and periodically throughout the program. Seven aliquots each of 27 samples were analyzed throughout Phase IIIA, and provisional MDLs were calculated equal to three times the standard deviation of each set of seven analyses. The Phase IIIA MDL was determined by averaging the MDLs of individual MDL test results. The Practical Quantitation Limit (PQL) is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The PQLs for arsenic and lead were calculated as the average of 10 times the standard deviation in each test sample. Values reported between the MDL and PQL are considered estimated concentrations.

Analyte	MDL (mg/kg)	PQL (mg/kg)
Arsenic	11	36
Lead	52	173

#### *Instrument Calibration*

The accuracy of the sample results was achieved through XRF instrument calibration and re-standardization, supplemented with:

- Daily energy calibration check
- Daily initial calibration verification through analysis of three or more Standard Reference Materials (SRM) with certified concentrations provided by the National Institute of Standards and Technology (NIST)
- Continuing calibration verification by analysis of one SRM with each analytical batch

The NIST SRM results are presented in Figures 9 through 12. If a NIST standard exceeded the control limit, then data for samples analyzed with that standard were rejected and the analytical batch was re-analyzed. A small number of NIST 2704 and NIST 2709 standards shown in Figure 9 exceed the final criteria because the criteria at the time of analysis was based on plus or minus one MDL, and the provisional arsenic MDL of 12 mg/kg was in use.

#### *Blind Standards*

Accuracy also is measured by submitting blind standards for analysis. These standards are contained and labeled in the same manner as field samples, and therefore the analyst cannot identify them as quality control standards. Nominal values for six lots (Lots A - F) were established through multiple analyses of subsamples from the lot. A slightly higher degree of variability is expected for the blind standards as compared to the NIST standards used in the calibration verification because the blind standards prepared for this program do not have certified concentrations and the matrix is more variable. The blind standards results are presented in Figures 13 through 16.

### **Representativeness**

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic or condition, and is achieved through proper design of a sampling program.

Representativeness of soil samples has been assessed through preparation and analysis of blanks, comparison of field duplicates, and intra-sample variability tests.

#### ***Instrument and Method Blanks***

Instrument blanks consisted of clean sand and were run with each analytical batch. Method blanks consisted of clean sand that was processed through the entire laboratory preparation and analytical procedures on a daily basis. Instrument and method blank results all were below the MDLs and demonstrate that cross contamination did not occur between samples in the field laboratory.

#### ***Rinse Blanks***

Rinse blanks were prepared by rinsing decontaminated soil sampling equipment (augers, trowels, and bowls) with deionized water and collecting the water for analysis. Rinse blanks were collected at a frequency of 3.5% of the field samples, which is less than the 5% (one for every twenty field samples) stated in the QAPP. However, neither arsenic nor lead were reported present in any of the one hundred seventy-four rinse blanks collected, which demonstrates effective decontamination of soil sampling equipment.

#### ***Field Duplicates***

Three field duplicates were collected of the garden soil samples and eleven field duplicates were collected from school yard samples. In garden soils, two of the arsenic and all three pairs of lead values were greater than the MDL. The relative percent difference between the original field sample and its duplicate for lead was very low ranging from 0% - 2%, while the relative percent differences for arsenic were 25% and 54%. In school yard samples, ten of the eleven arsenic concentrations were below the MDL and the single reported value exhibited a relative percent difference of 42%. Ten of the eleven samples contained lead at less than five times the MDL and met the criteria of less than one MDL difference between the original field sample and its duplicate. One sample was subject to relative percent difference criteria for lead and met the criteria at 14%.

#### ***Variability Tests***

Intra-sample variability tests were performed to verify that homogenization of the composite sample was sufficient to reduce variability, which ensures that the portion that is prepared and analyzed is representative of the composite (and therefore representative of the property).

Variability tests involved collecting and separately preparing seven aliquots of the homogenized composite sample. Tests were performed on ten samples. For concentrations that were greater than the MDL, all test samples exhibited a percent relative standard deviation of less than 25%.

### **Comparability**

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. Comparability was evaluated during Phase III through preparation and analysis of confirmation soil samples.

### *Confirmation Samples*

A percentage of the samples were split and prepared as confirmation samples. Initially, one confirmation sample was prepared for every three field samples, and after initial results were reviewed, the frequency was reduced to one in ten field samples. The confirmation samples were submitted to an off-site, fixed laboratory for analysis by EPA Method 6010B, Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP). A portion of the confirmation sample results were qualified as estimated based on quality control data. However, no major anomalies were identified and no data were rejected. The comparison of XRF versus ICP results are presented in Figure 17, which exhibits a high degree of correlation (with the exception of two lead values) and documents that the XRF results are generally comparable to those from ICP analysis.

### **Completeness**

Completeness is a measure of the percent of useable data generated as compared to the data required and collected. Surface soil samples were collected from 1550 residential properties, which is 100% of the properties for which yards were physically accessible, and 98% of the properties for which written consent for access was received. Useable data were produced for 100% of the samples collected. These achievements are consistent with the project completeness goals of sampling 100% of properties granting access and generating useable data for greater than 90% of the data generated.

## **DUST SAMPLE DATA QUALITY**

Dust samples were collected from indoor flooring surfaces using a high volume vacuum sampler. Candidate homes were identified based on a stratified random analysis and resident consent for interior access. One composite dust sample per home was collected from each of 76 homes. Dust samples were prepared and analyzed at an off-site, fixed laboratory by ICP using EPA Method 6010B.

### **Precision**

Matrix spike duplicates and laboratory control sample duplicates were analyzed to measure precision of dust sample analyses.

#### *Matrix Spike Duplicates*

Matrix spike duplicates were prepared and analyzed at the required frequency of one per batch. The relative percent difference between the original matrix spike sample and its duplicate ranged from 0% to 2% for arsenic and 1% to 3% for lead, which meets the criteria of less than 25% relative percent difference.

#### *Laboratory Control Sample Duplicates*

Laboratory control sample duplicates were prepared and analyzed at the required frequency of one per batch. The relative percent difference between the original laboratory control sample and its duplicate ranged from 0% to 1% for arsenic and 1% to 2% for lead, which meets the criteria of less than 25% relative percent difference.

### **Accuracy**

The accuracy of the dust sample results was verified through the initial and continuing calibrations, matrix spike samples, laboratory control samples, interference check samples, and blind standards.

#### ***Instrument Calibration***

Initial calibration verification demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run, while continuing calibration verification demonstrates that the initial calibration is still valid. Initial and continuing calibration verifications met the quality control criteria for percent recovery of 90-110% of the certified standard concentration, ranging from 97% to 103%. The initial calibration verifications were analyzed at the beginning of each analytical run. The continuing calibration verification was analyzed every ten samples.

#### ***Matrix Spikes***

Matrix spikes are prepared by adding a known concentration of one or more analytes to a field sample, and is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. All matrix spikes met the quality control criteria for percent recovery of 75-125%. Recoveries ranged from 97% to 101% for arsenic and 68% to 111% for lead (the 68% recovery did not result in data qualification because the spike concentration was less than four times the sample concentration). Matrix spikes were analyzed at a frequency of one per batch.

#### ***Laboratory Control Samples***

The laboratory control sample serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Laboratory control samples were analyzed at the proper frequency and met the acceptance criteria for percent recovery of 80-120%, ranging from 100% to 104% for arsenic and 94% to 99% for lead.

#### ***Interference Check Samples***

Interference check samples verify the laboratory's inter-element and background correction factors. The interference check sample was analyzed at the beginning and end of each analytical run. The percent recovery results were within the quality control limits of 80-100%, ranging from 96% to 103% for arsenic and 87% to 94% for lead.

#### ***Blind Standards***

Four aliquots of four samples were prepared and sent to two separate laboratories to determine the nominal concentration of each sample. The nominal concentration was determined by averaging the eight reported values for arsenic and for lead. Thirty blind standards were then submitted for analysis along with the field dust samples. The results of blind standards analyses are presented in Figures 18 and 19.

### **Representativeness**

Representativeness of dust samples has been assessed through instrument blanks, method blanks, and rinse blanks.

#### *Instrument and Method Blanks*

None of the initial or continuing calibration blanks contained arsenic or lead above the reporting limit. The initial calibration blank was analyzed at the beginning of each analytical run and continuing calibration blanks were analyzed after every ten samples. One method blank was analyzed per batch. Method blanks also were free from contamination, indicating that laboratory contamination did not occur.

#### *Rinse Blanks*

Rinse blanks were collected at the specified frequency of 5% (one for every twenty field samples) by rinsing the interior surfaces of the decontaminated vacuum sampler that contact the dust sample with deionized water and containing the rinsate. The rinse blanks were free from contamination, which demonstrates that proper decontamination was performed to reduce the possibility of cross contamination.

#### **Completeness**

Completeness is expressed as the percent of usable data as compared to the data collected. Unusable data are those results reported by the laboratory but rejected during the data validation process. Objectives for dust sampling included collecting between 60 and 90 samples. The desired quantity was achieved and 100% percent of the dust data are useable.

### **VEGETABLE SAMPLE DATA QUALITY**

Properties where gardens had been documented and that still had vegetables available in October were sampled for vegetables prior to hard frost. A total of 72 vegetable samples were collected from 19 gardens. Vegetables were prepared and analyzed at an off-site, fixed laboratory by ICP-MS using EPA Method 6020.

#### **Precision**

Matrix spike duplicates were analyzed to measure precision of vegetable sample analyses.

#### *Matrix Spike Duplicates*

Matrix spike duplicates were prepared and analyzed at the required frequency of one per batch. The relative percent difference between the original matrix spike sample and its duplicate ranged from 0% to 2% for arsenic and 1% to 3% for lead, which meets the criteria of less than 25% relative percent difference.

#### **Accuracy**

The accuracy of the dust sample results was verified through the initial and continuing calibrations, matrix spike and post digestion spike recoveries, interference check samples, laboratory control samples an MDL study, and blind standards.

#### *Instrument Calibration*

Initial and continuing calibration verifications met the quality control criteria for percent recovery of 90-110%, ranging from 97% to 103%. The initial calibration verifications were analyzed at

the beginning of each analytical run. The continuing calibration verification was analyzed every ten samples.

#### *Matrix Spikes*

All matrix spikes and post digestion spikes met the quality control criteria for percent recovery of 75-125% and frequency criteria of one per batch, ranging from 106% to 108% for arsenic and 87% to 97% for lead.

#### *Interference Check Samples*

Interference check samples were analyzed at the beginning and end of each analytical run. The interference check sample percent recovery results were within the quality control limits of 80-120% at 99% for arsenic (lead was below the MDL).

#### *Laboratory Control Samples*

All laboratory control samples met the quality control criteria established for the standard reference materials used, ranging from 73% to 107% for arsenic and 88% to 98% for lead.

#### *Method Detection Limit Study*

Seven aliquots of one sample (NIST SRM 1570, spinach leaves) were prepared and analyzed individually. The SRM certified value for arsenic is 0.068 mg/kg (plus or minus 0.012 mg/kg) and the uncertified value for lead is 0.2 mg/kg. Results of seven analyses of the SRM exhibited a low standard deviation (less than 0.012) for both analytes, which documented that the targeted method detection limit of 0.05 mg/kg was achieved.

#### *Blind Standards*

NIST SRM 1570 was used as to prepare blind standards submitted to the laboratory along with the vegetable samples. Measured concentrations ranged from 74% to 162% of the certified value for arsenic and were 80% of the non-certified value for lead. The SRM arsenic and lead concentrations are near the MDL and less than the PQL, and therefore reported values in this range are considered estimated.

#### **Representativeness**

Representativeness has been assessed through instrument blanks and method blanks.

#### *Instrument and Method Blanks*

None of the initial or continuing calibration blanks contained arsenic or lead above the reporting limit. Method blanks were also free from contamination, indicating that laboratory contamination did not occur. The method blanks were analyzed at the correct frequency of one per batch. The initial calibration blank was analyzed at the beginning of each analytical run and continuing calibration blanks were analyzed after every ten samples.

#### **Completeness**

No vegetable sample results were rejected upon validation of the data, and therefore 100% percent of the vegetable data are useable.



## PROPERTY SOIL DISTRIBUTIONAL ANALYSIS

In the "Project Plan for the Vasquez Boulevard & I-70 Site, Denver, Colorado, Phase III Field Investigation" (ISSI, 8/4/99), Appendix D, "Screening Level Evaluation of Risks from Acute and Subchronic Exposure to Arsenic in Soil" sets forth a three tiered decision rule that the sampling results from a residential property must pass in order for the property to be considered below acceptable risk levels. These are:

- 1) 95% upper confidence limit (UCL) of three composites < RBCchronic
- 2) Maximum composite value  $\leq$  MTCVacute
- 3) Maximum composite value  $\leq$  MTCVsubchronic

A key assumption for test number one that is the data are normally distributed. Whereas statistical tests (USEPA, DataQuest software) are available to determine whether sample data can be considered normally distributed, it is difficult to determine accurately whether three individual samples are in fact normally distributed. Given this, the field sampling program used a composite sampling design. The composite design was implemented in an attempt to ensure normal data from each residential property.

Monte Carlo simulations were run to determine a reasonable and reliable number of samples that should be combined into a single composite sample (ISSI 8/4/99). As a result of the simulation exercise, it was decided that ten samples would be combined to form a composite sample.

One of the early steps in the Data Quality Assessment process is to determine if sample data can be considered a normal so that a UCL on the mean can be calculated using normal statistics. Traditional quantitative tests for normality are not appropriate due to a paucity of data; therefore, other qualitative evaluations were performed to assess the assumption of normality.

If the field data from the sampled residential properties are to be considered normal, they should exhibit certain statistical characteristics. Among these include:

- 1) The coefficient of variation (CV) for the sample data should generally be in the range of the test data;
- 2) The CV should be below 1.0; and,
- 3) The maximum concentration observed at a residential property should not exceed the 95% UCL at more than 5% of the properties.

To test the statistical characteristic number one, the CVs from 901 properties were calculated and compared to the CVs in the simulated data, which ranged from 0.16 to 0.37 with associated means ranging from approximately 50ppm to 500ppm. The CVs from the sampled properties with sample concentrations above the method detection limit generally fell within this range.

For statistical characteristic number two, if a CV exceeds 1.0, the data are generally considered to be non-normally distributed (USEPA 1996, Guidance for Data Quality Assessment, QA/G-9). A total of 25 residential properties exhibited CVs in excess of 1.0. This is less than 2% of the residential properties and are attributable to concentrations either near the method detection limit or relatively high.

To test statistical characteristics number three, the maximum concentration at each residential property was compared to the mean plus two standard deviations, which encompass more than 95% of the sample distribution. No maximum sample concentration at any of the properties exceeded the mean plus two standard deviations. This provides an indication that the data from the residential properties do not violate the normality assumption.

In summary, the statistical characteristics of the sample data collected from the residential properties provide strong evidence that the sample data are normally distributed. Exceptions are restricted to very low and high concentrations, which should not impair decision making with regard to risk management.

**Table 1**  
**PHASE IIIA SOIL SAMPLING**  
**ANALYTICAL PROGRAM SUMMARY**

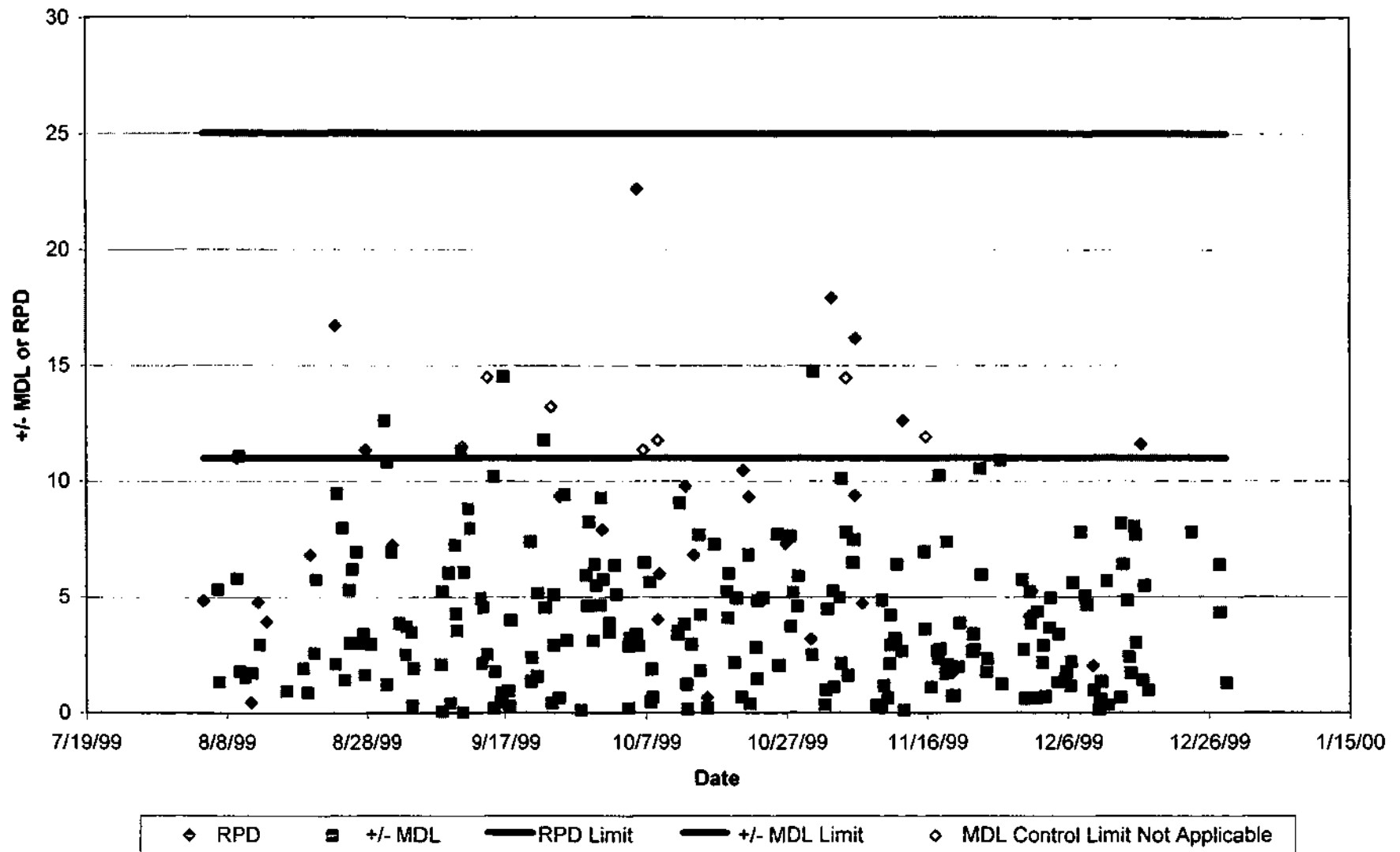
Field Samples	5207
Blind Duplicates	254
Lab Duplicates	264
Blind Standards	90
Lab Control Sample (SRM)	961
Instrument Blanks	415
Method Blanks	90
MDL Study Samples	27
Proficiency Samples	92
Variability Test Samples	72
Other Test Samples	118
Off-Site Confirmation Samples	751
<b>TOTAL</b>	<b>8341</b>

# Color Chart(s)

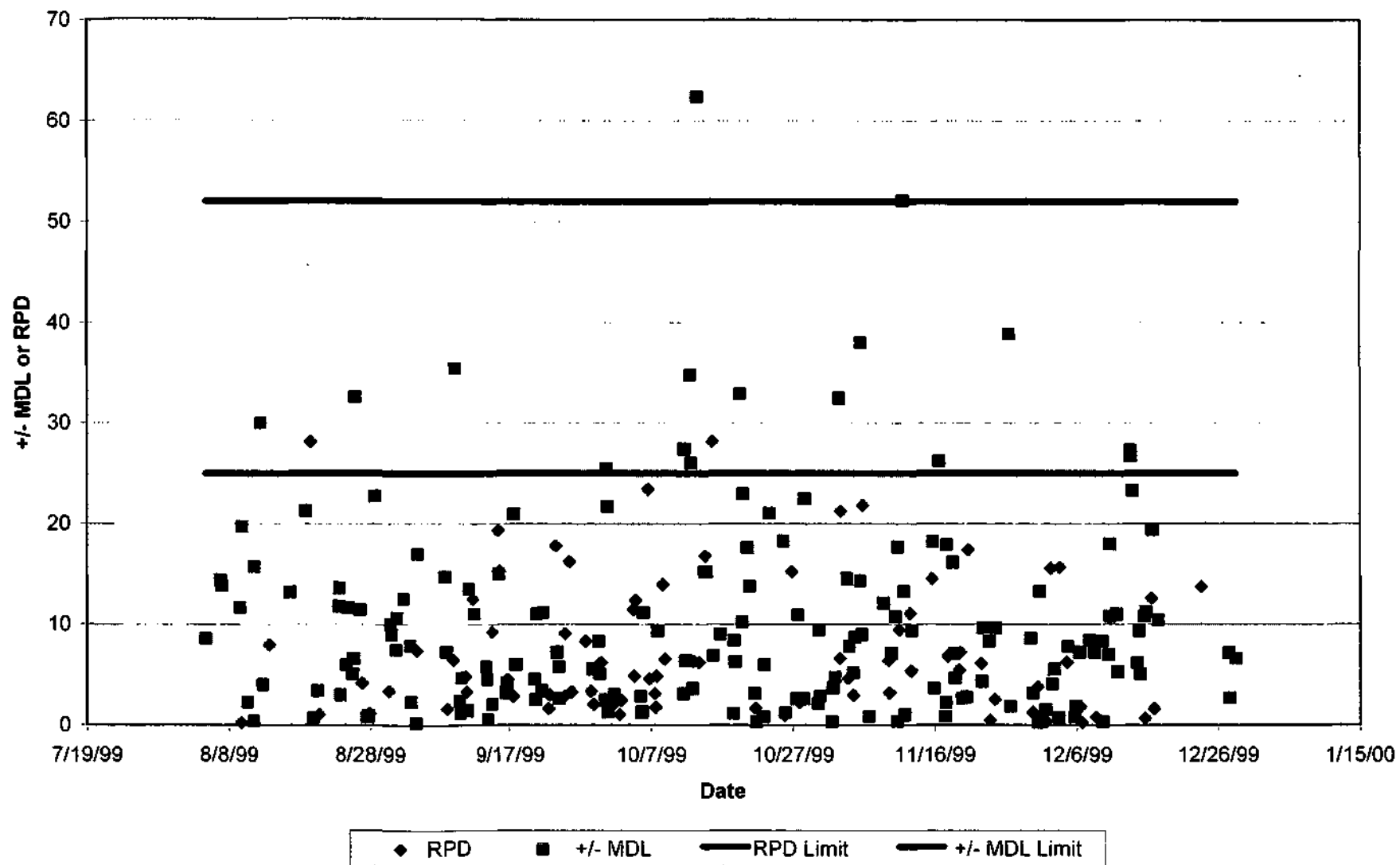
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contact the Superfund Records  
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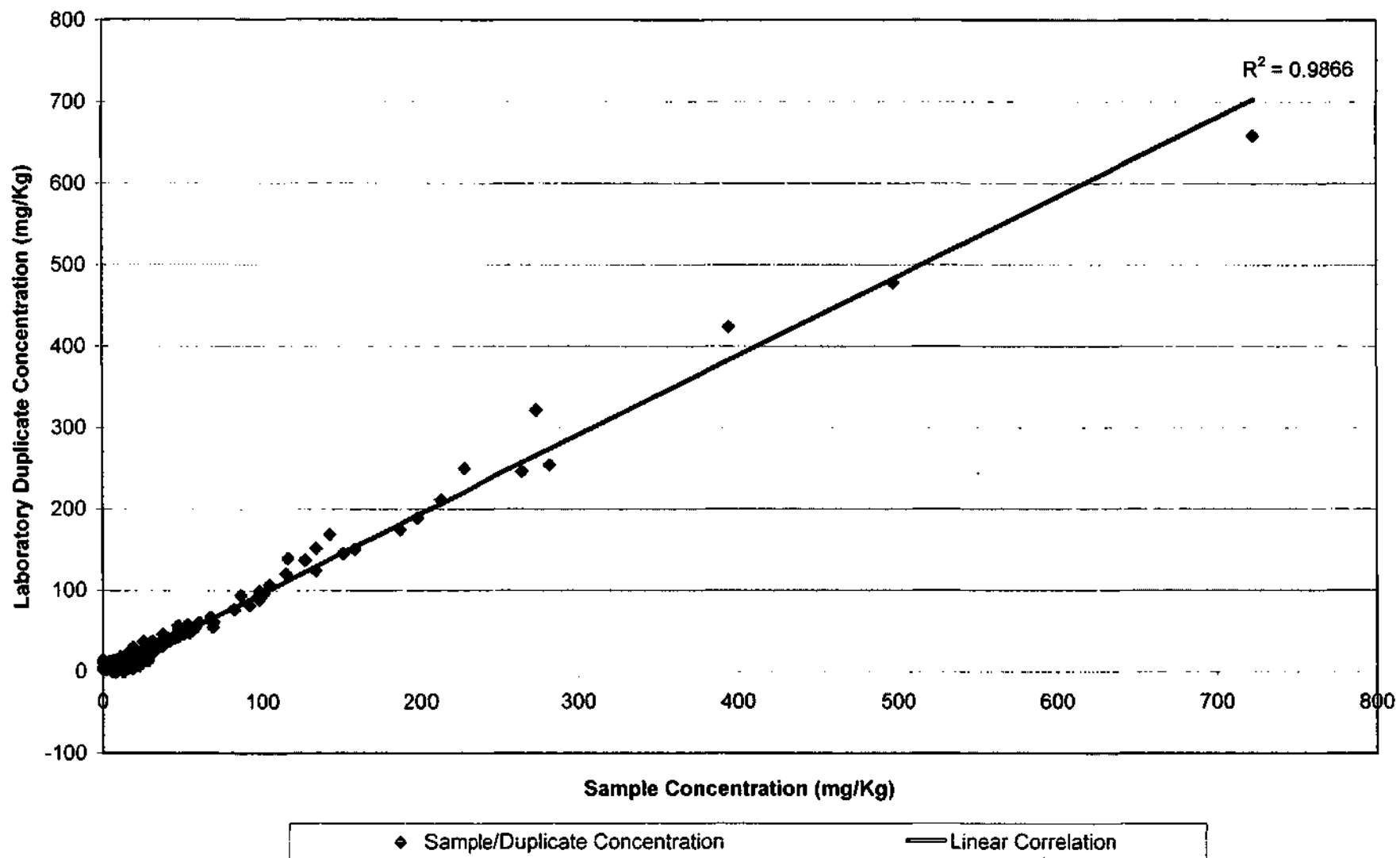
**Figure 1**  
**Laboratory Duplicate Results - Arsenic**



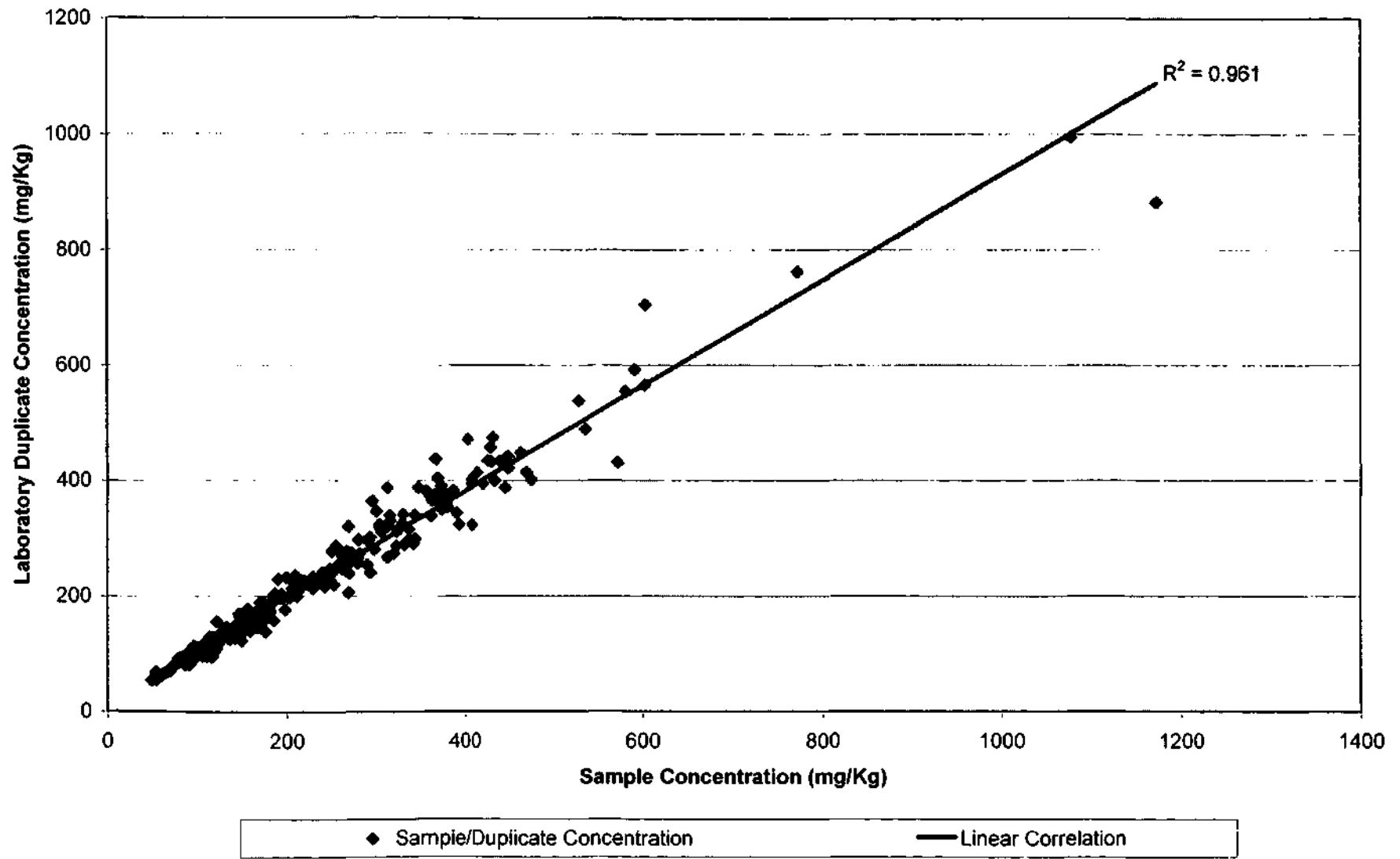
**Figure 2**  
**Laboratory Duplicate Results - Lead**



**Figure 3**  
**Laboratory Duplicate Correlation - Arsenic**

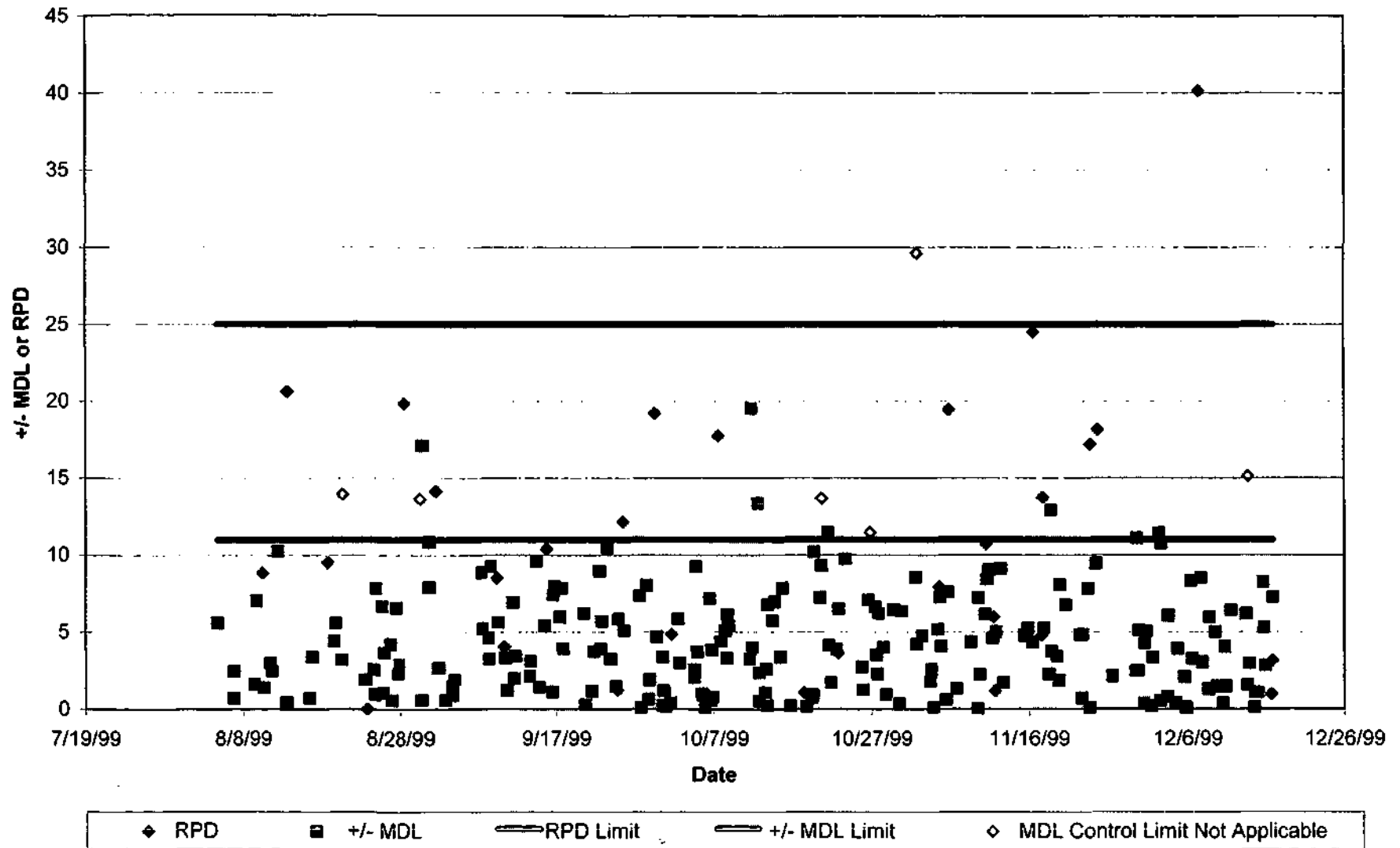


**Figure 4**  
**Laboratory Duplicate Correlation - Lead**

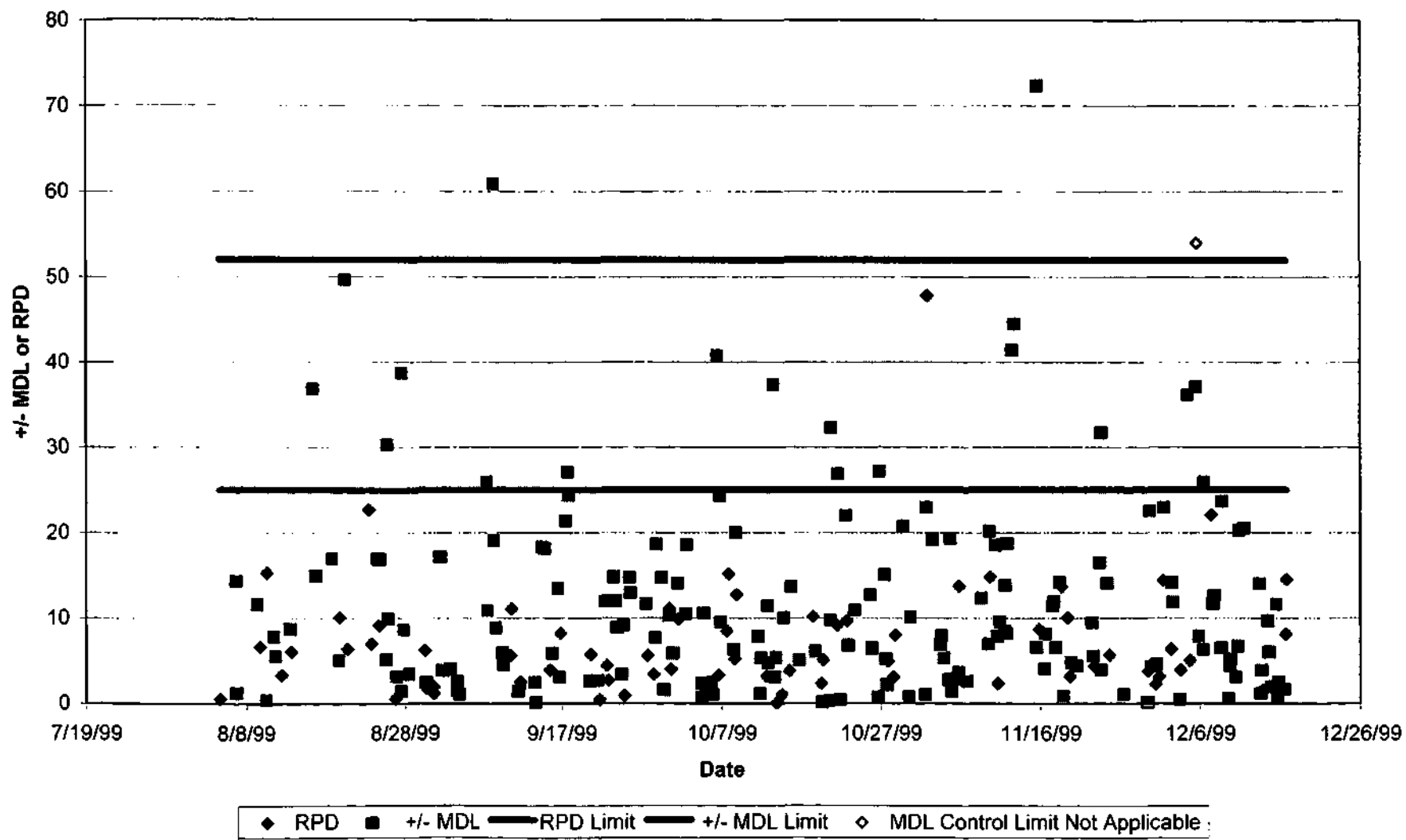




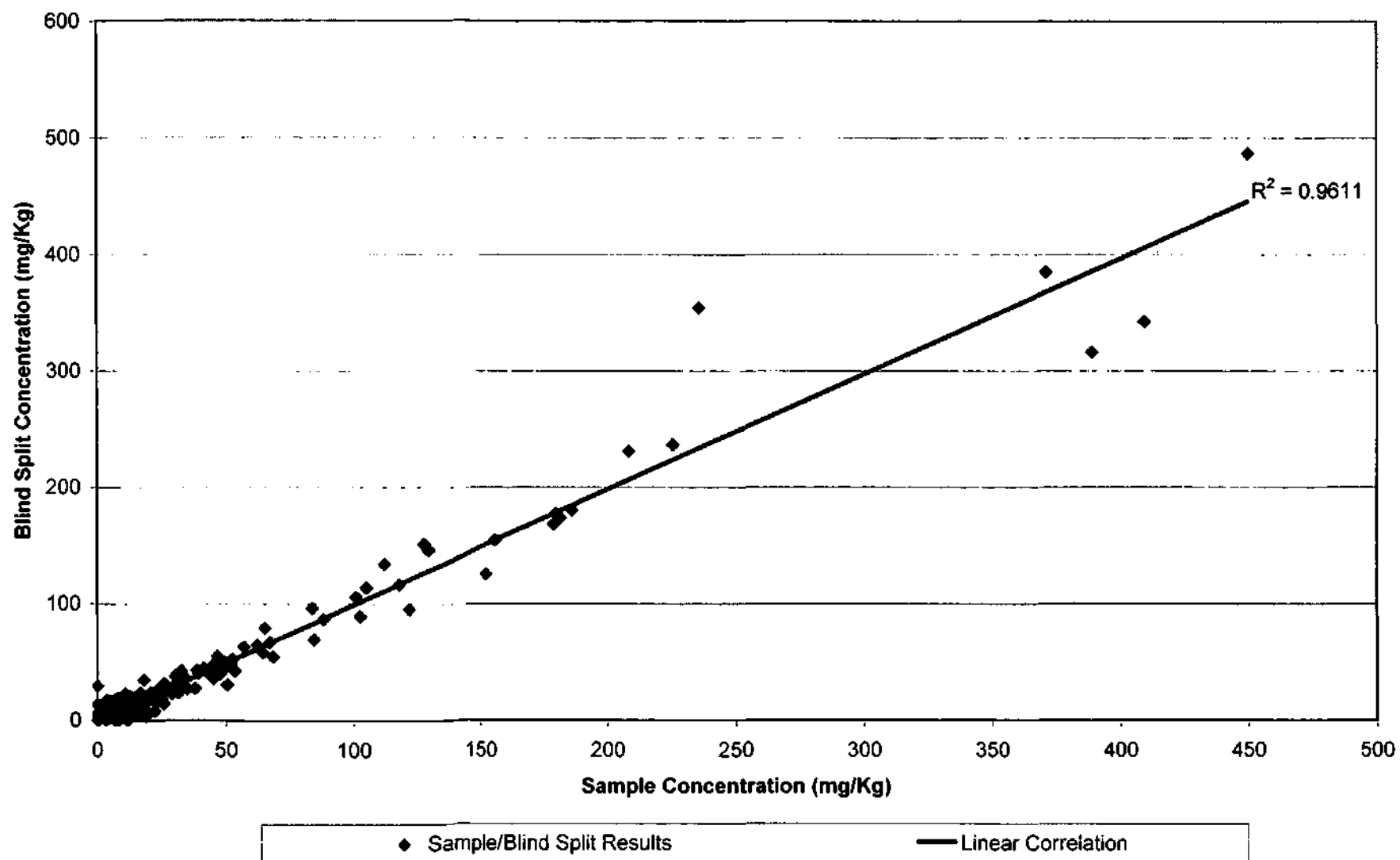
**Figure 5**  
**Blind Split Results - Arsenic**



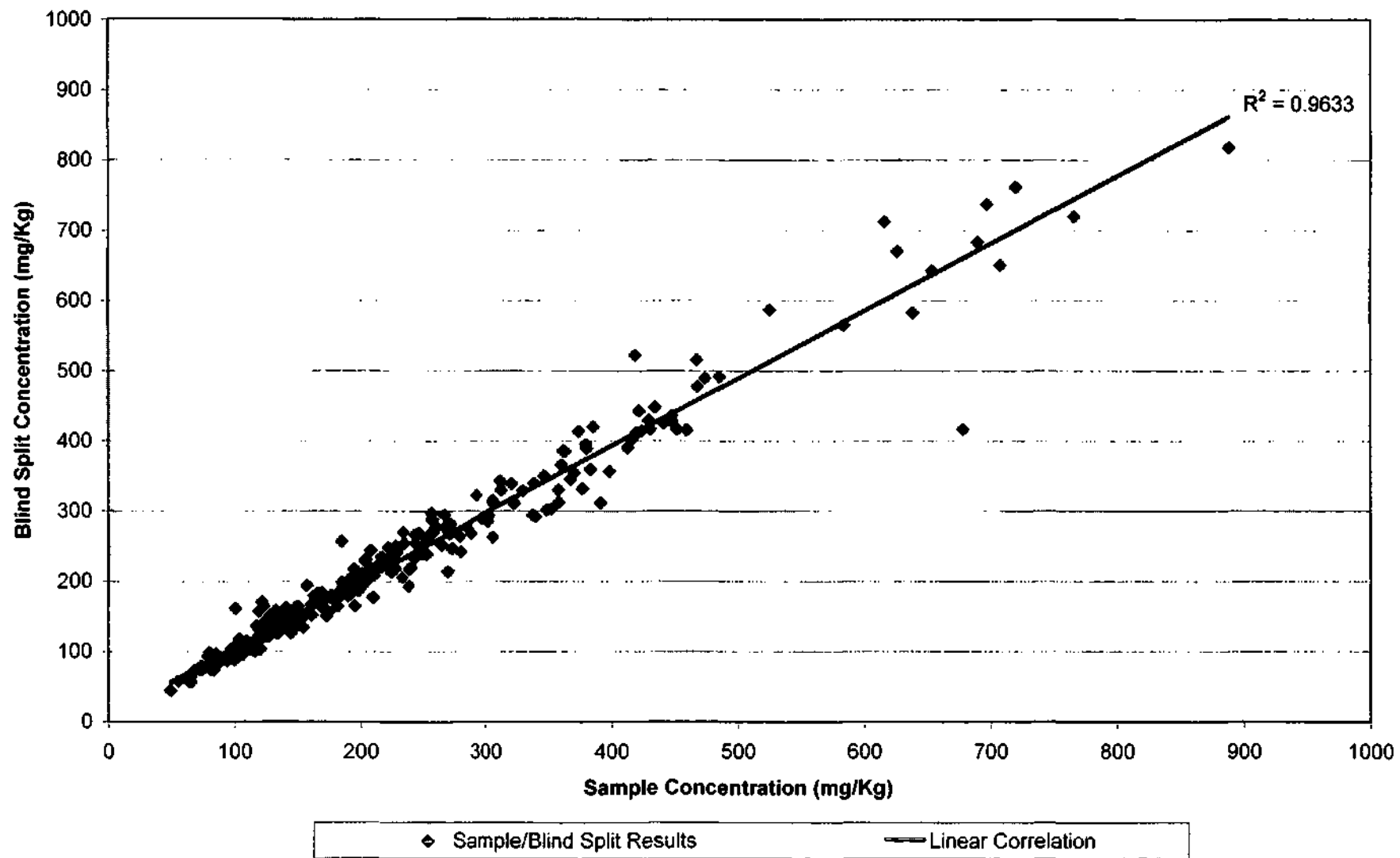
**Figure 6**  
**Blind Split Results - Lead**



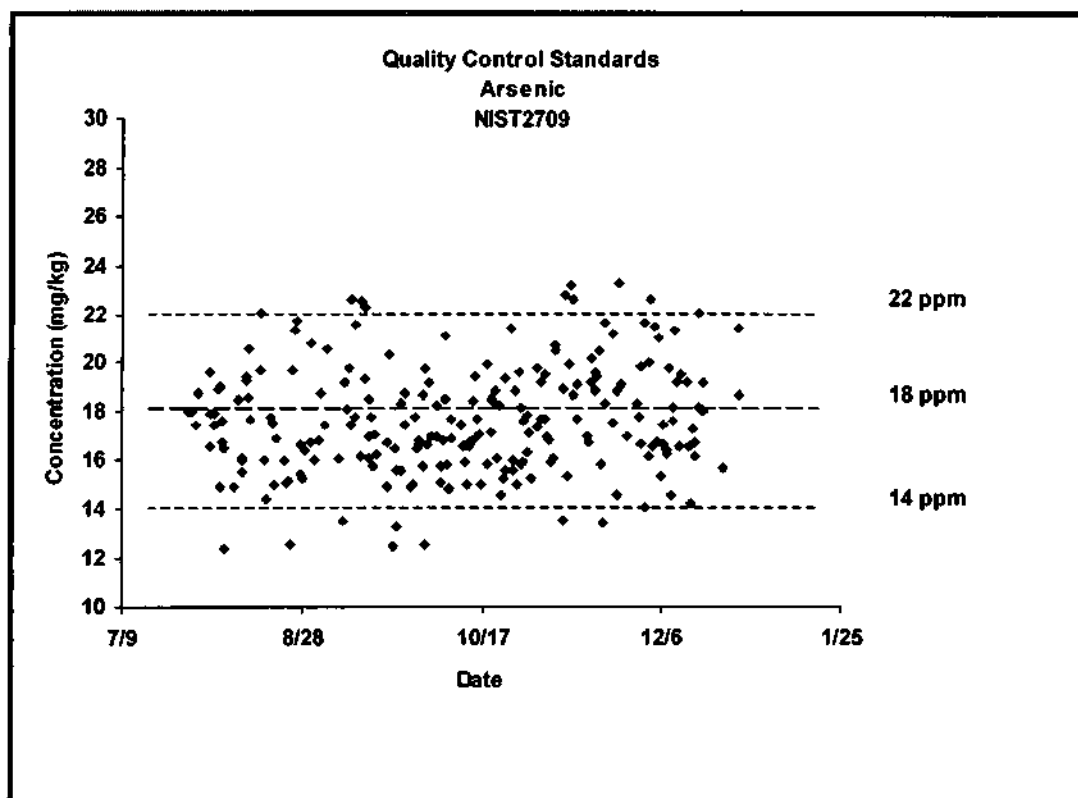
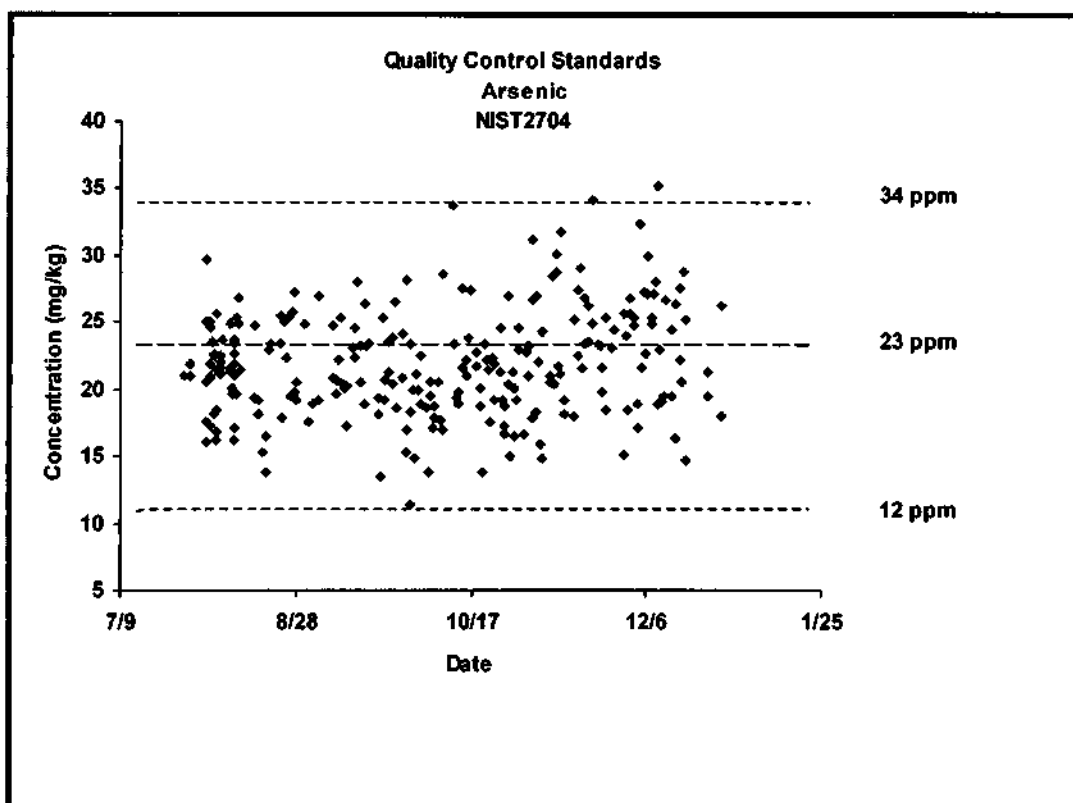
**Figure 7**  
**Blind Split Correlation - Arsenic**



**Figure 8**  
**Blind Split Correlation - Lead**



**Figure 9**



**Figure 10**

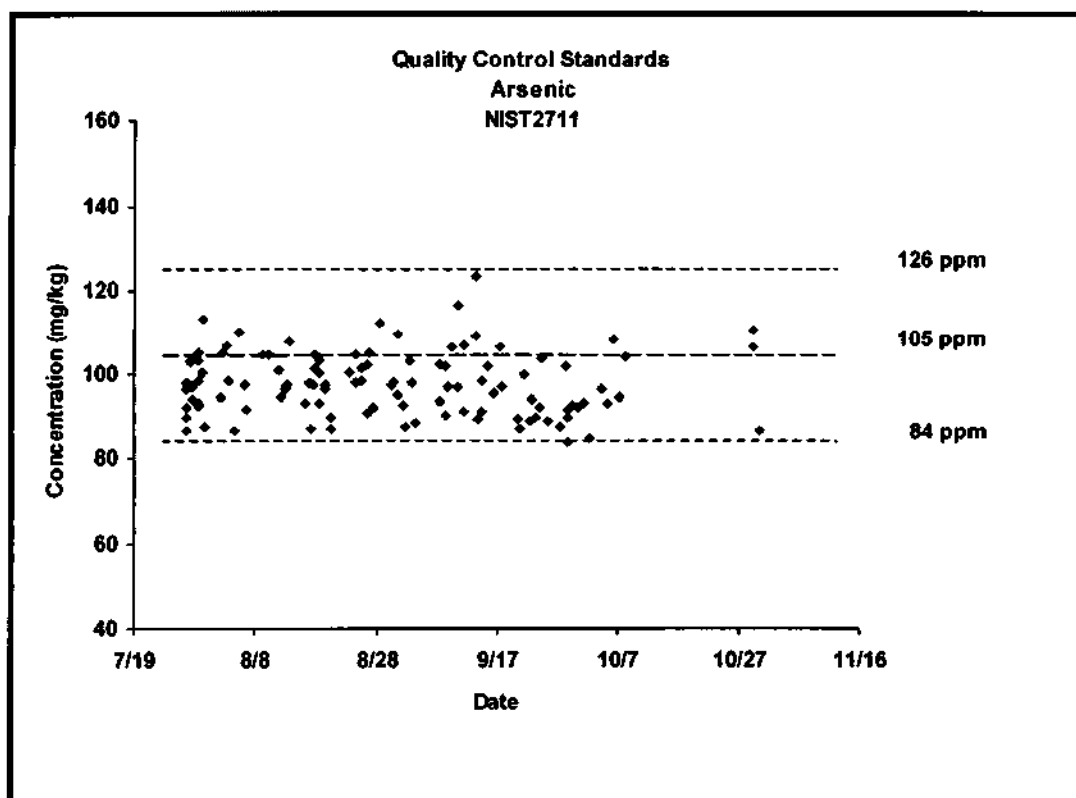
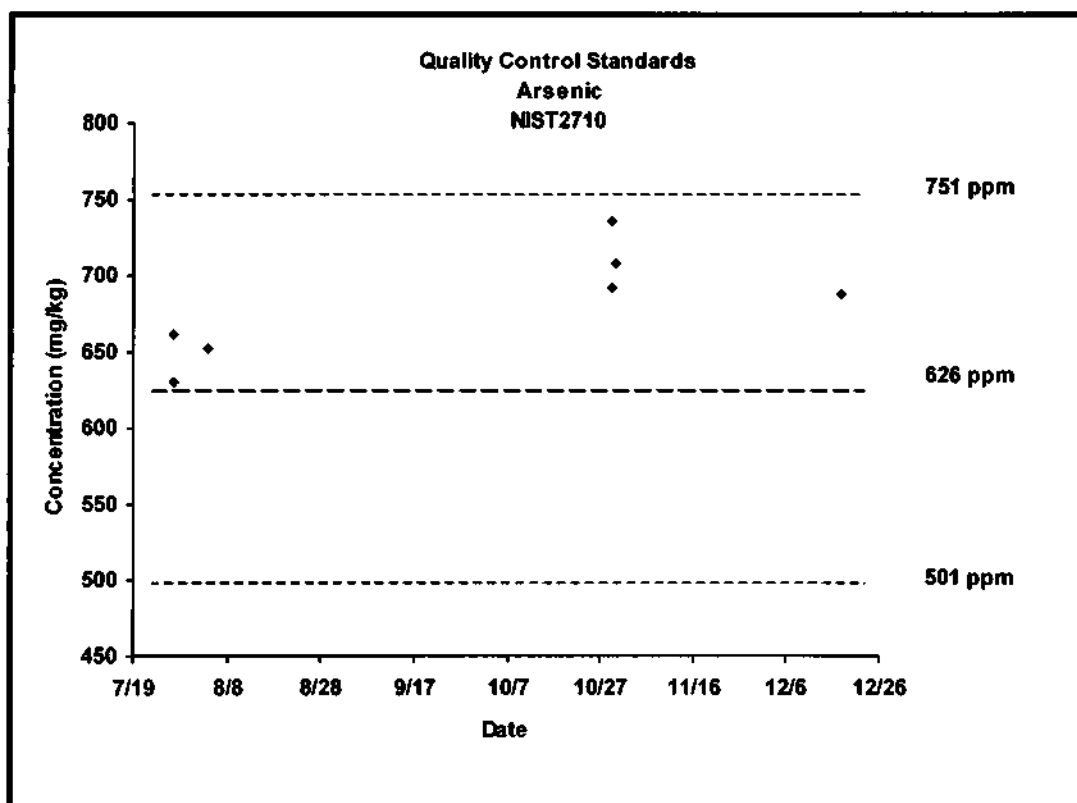
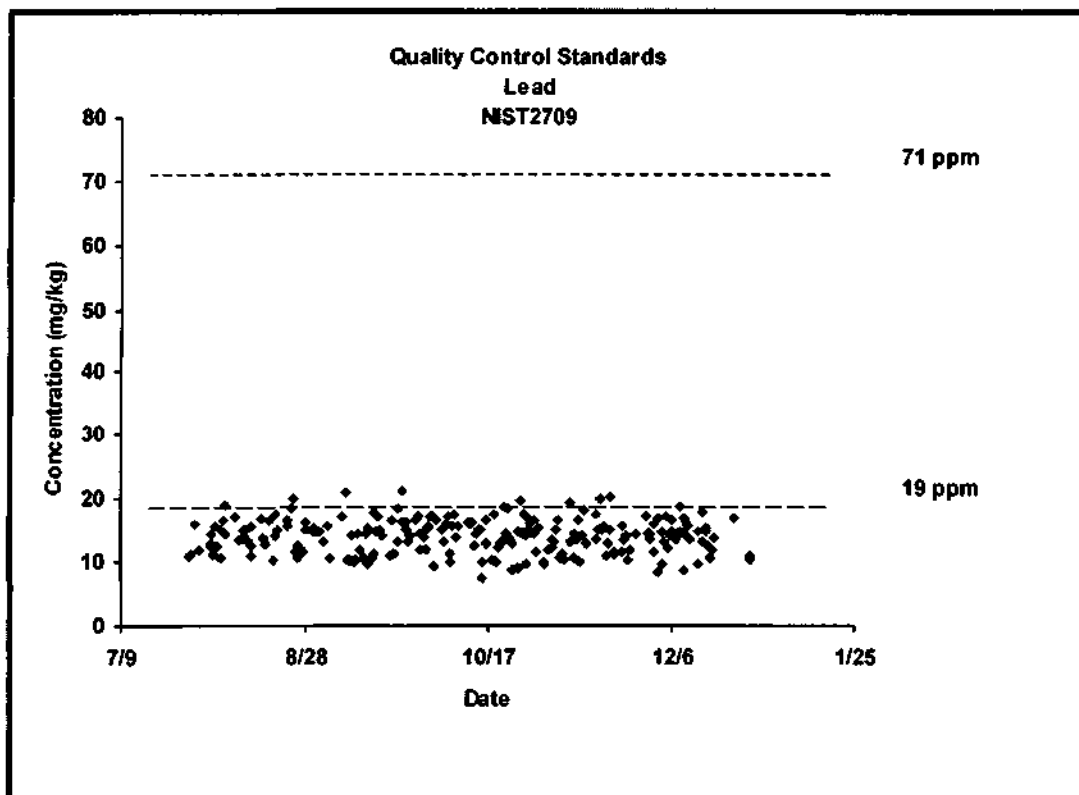
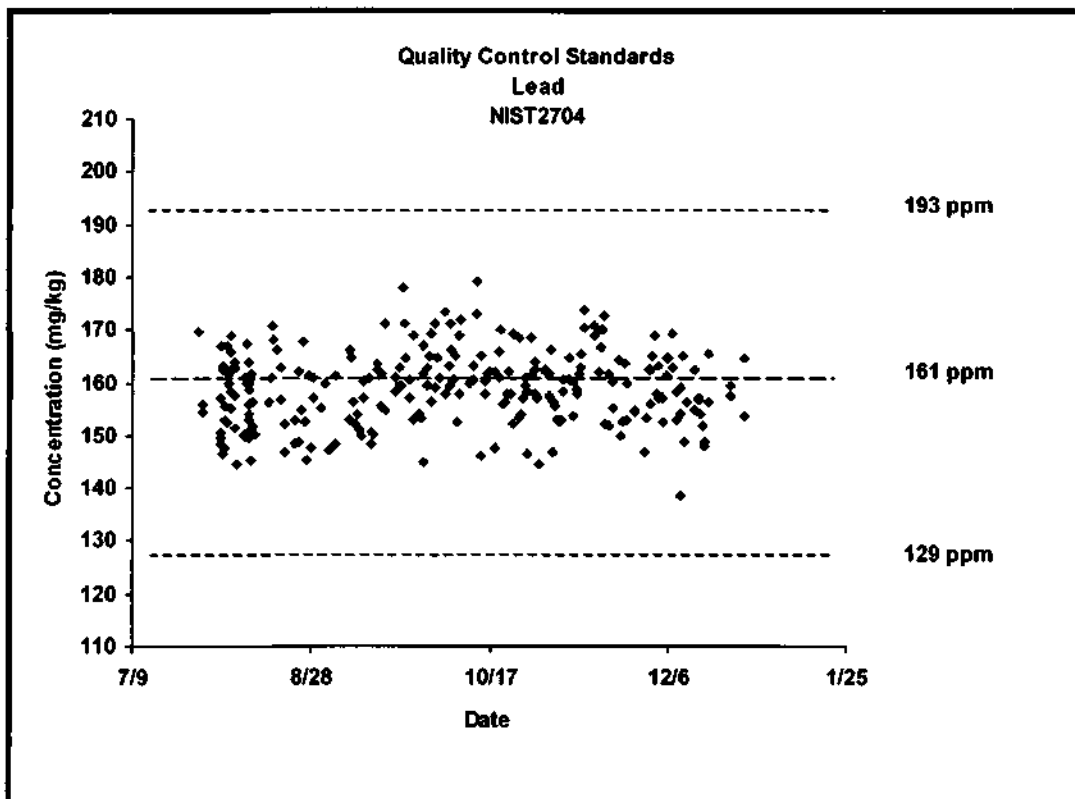
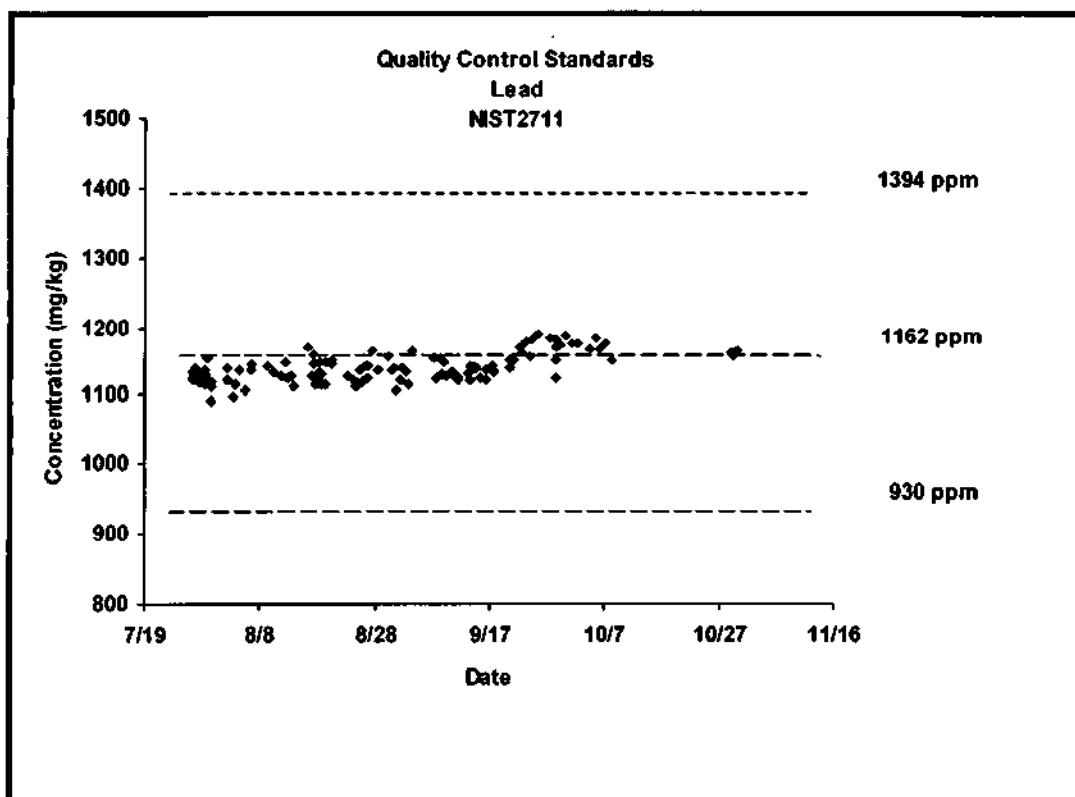
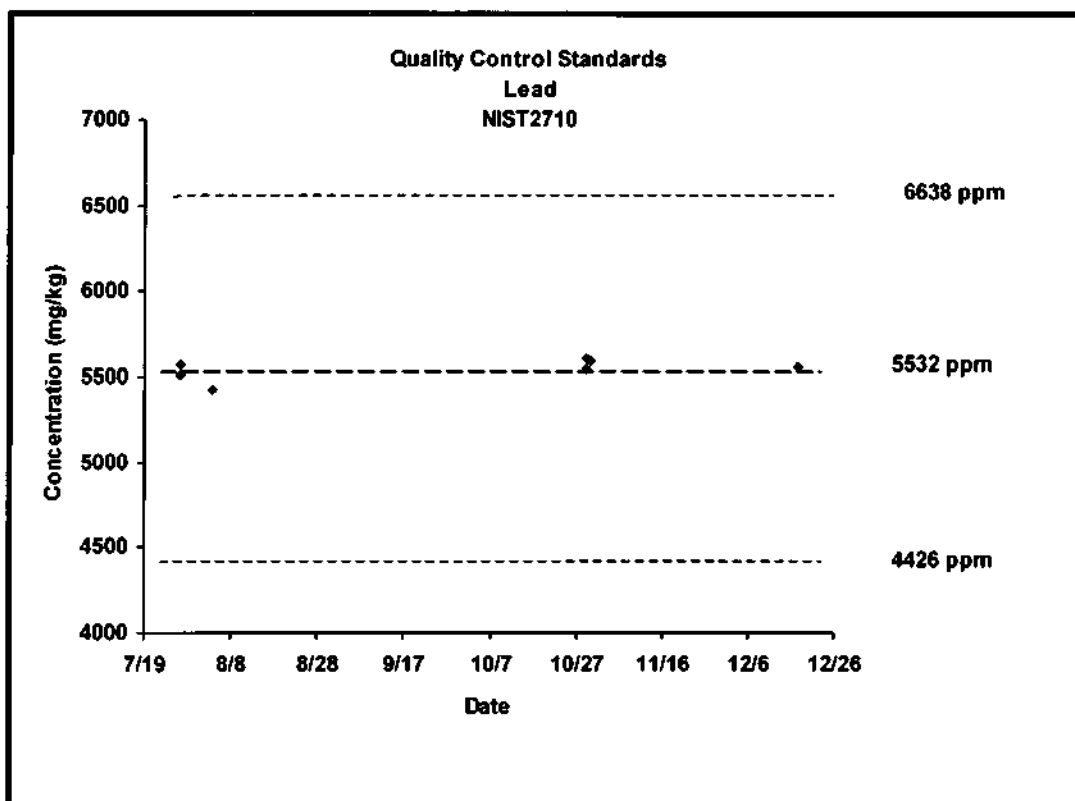


Figure 11

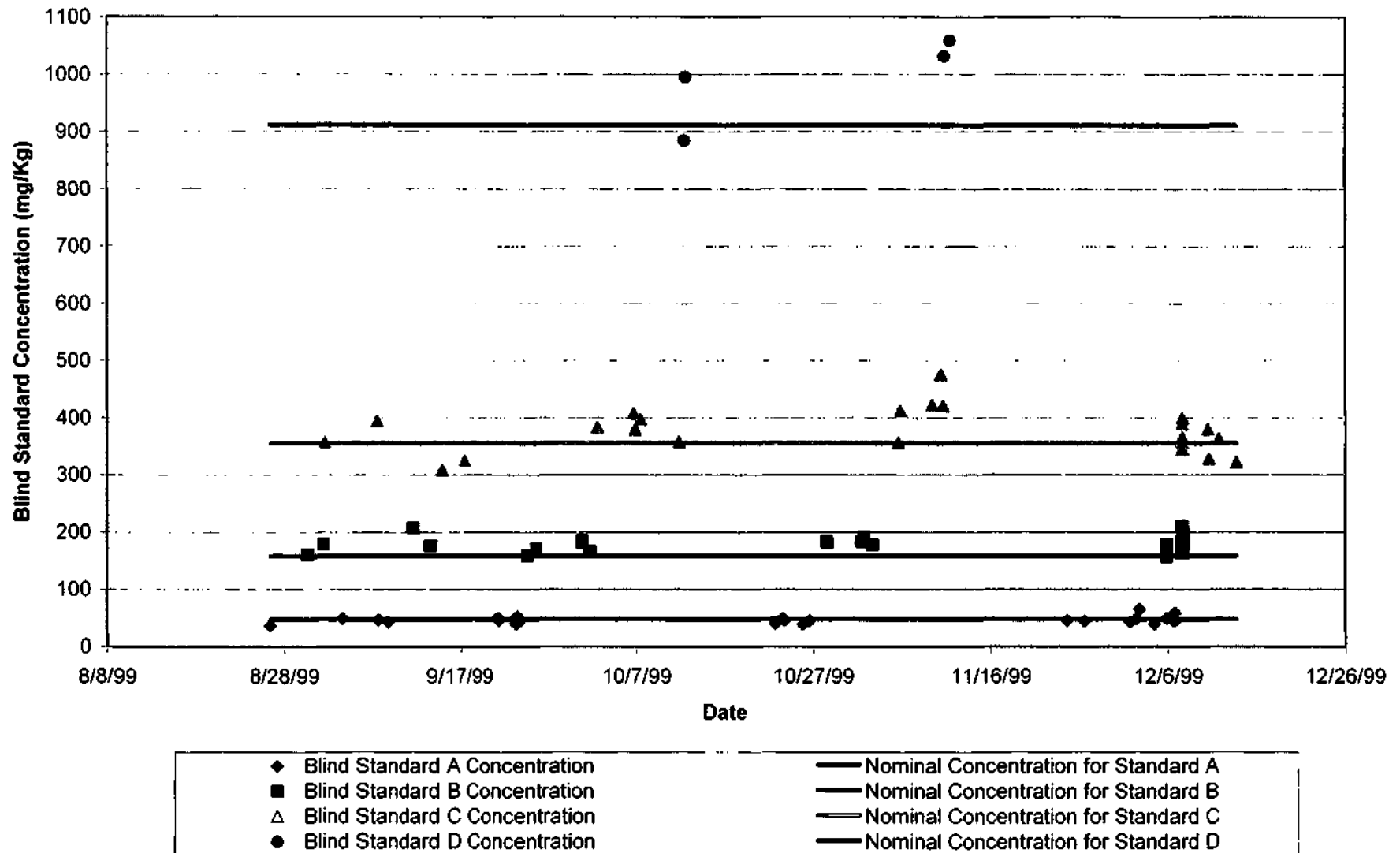


**Figure 12**

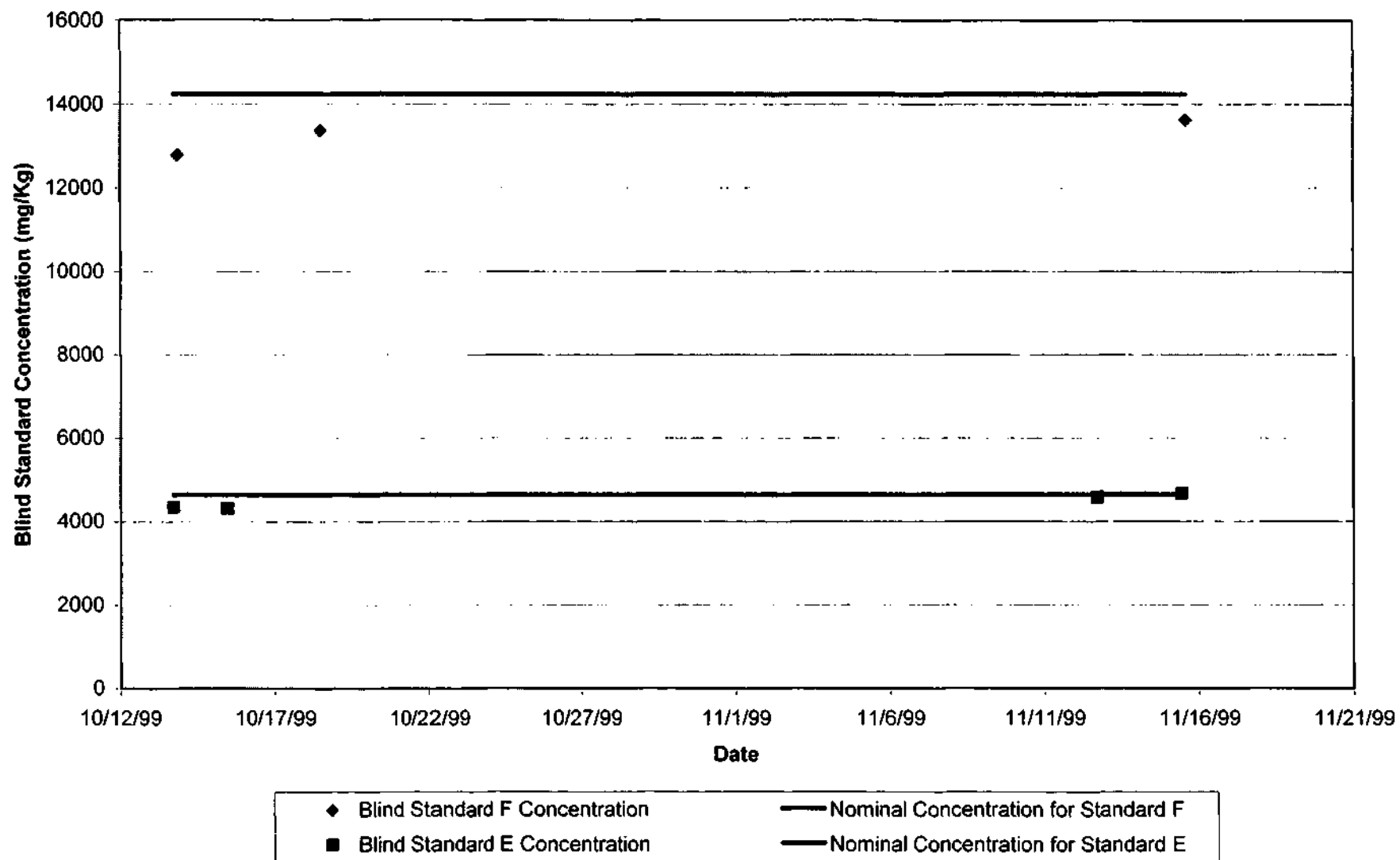




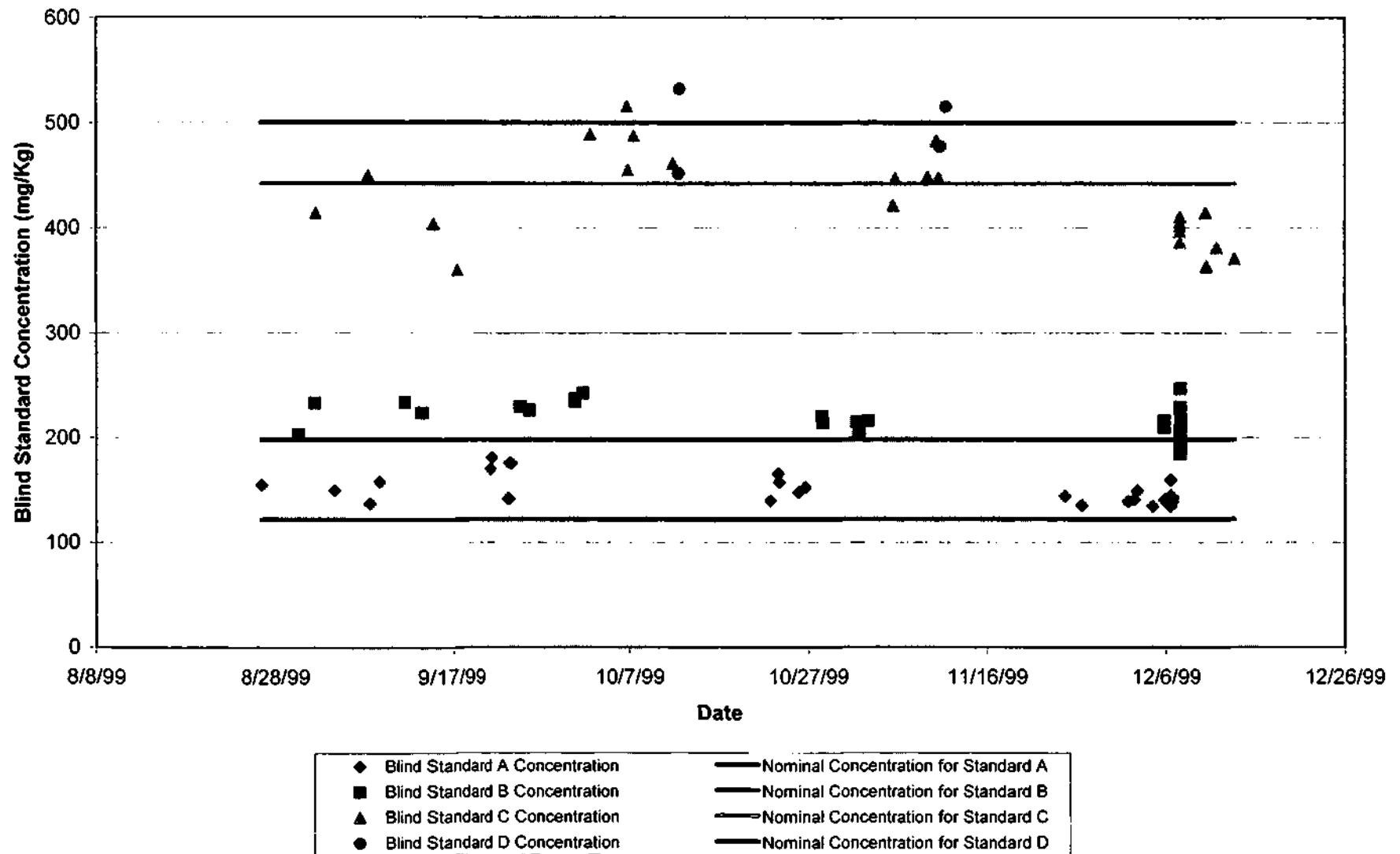
**Figure 13**  
**Blind Standards A, B, C, and D - Arsenic**



**Figure 14**  
**Blind Standards E and F - Arsenic**



**Figure 15**  
**Blind Standards A, B, C, and D - Lead**



**Figure 16**  
**Blind Standards E and F - Lead**

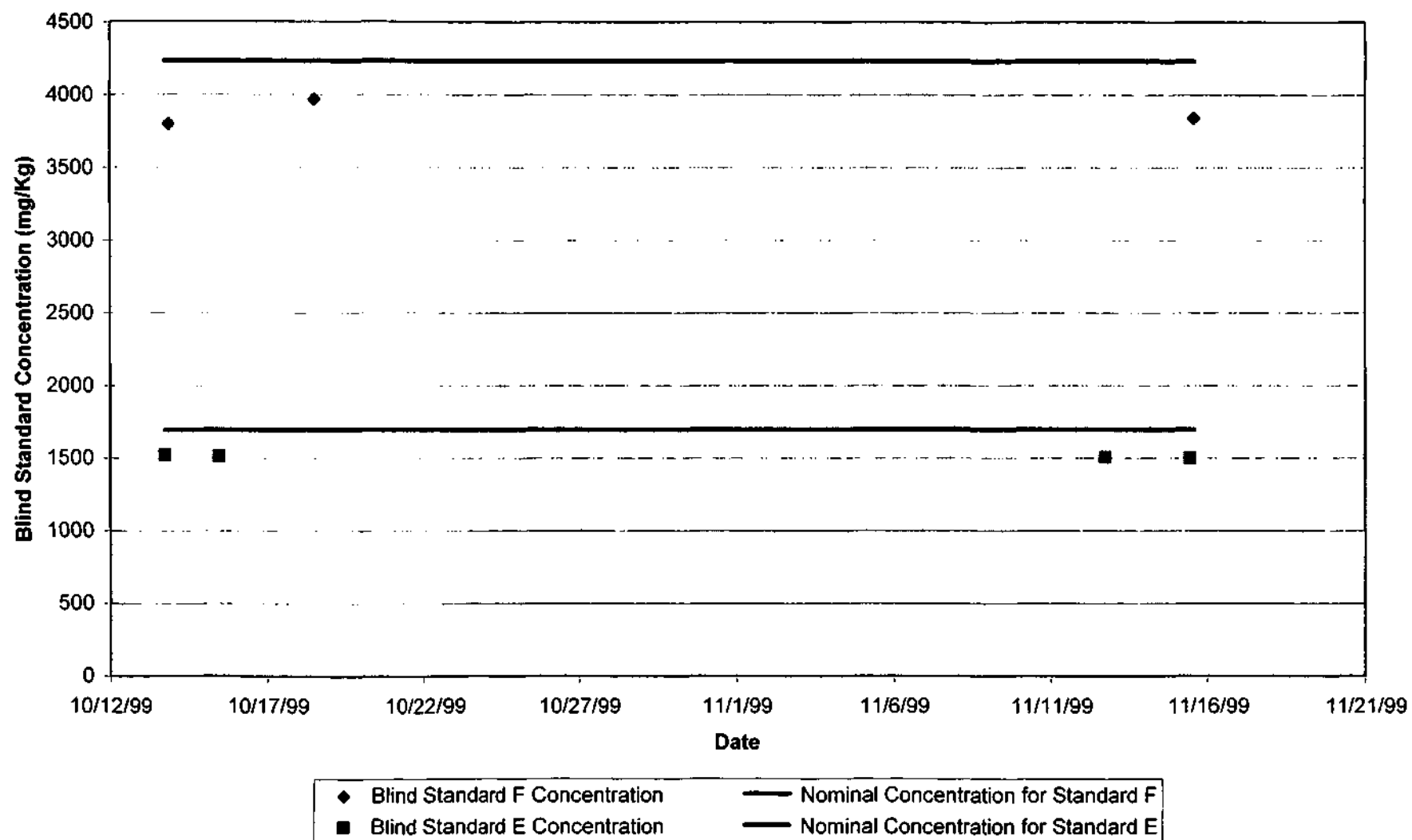
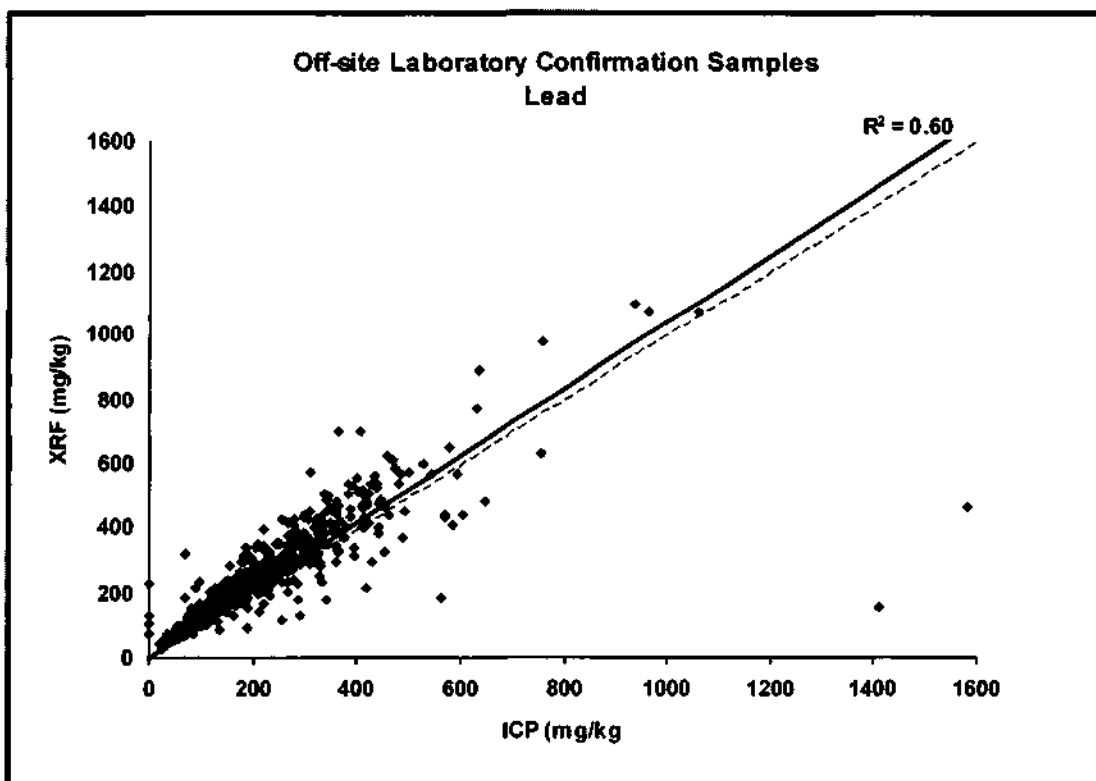
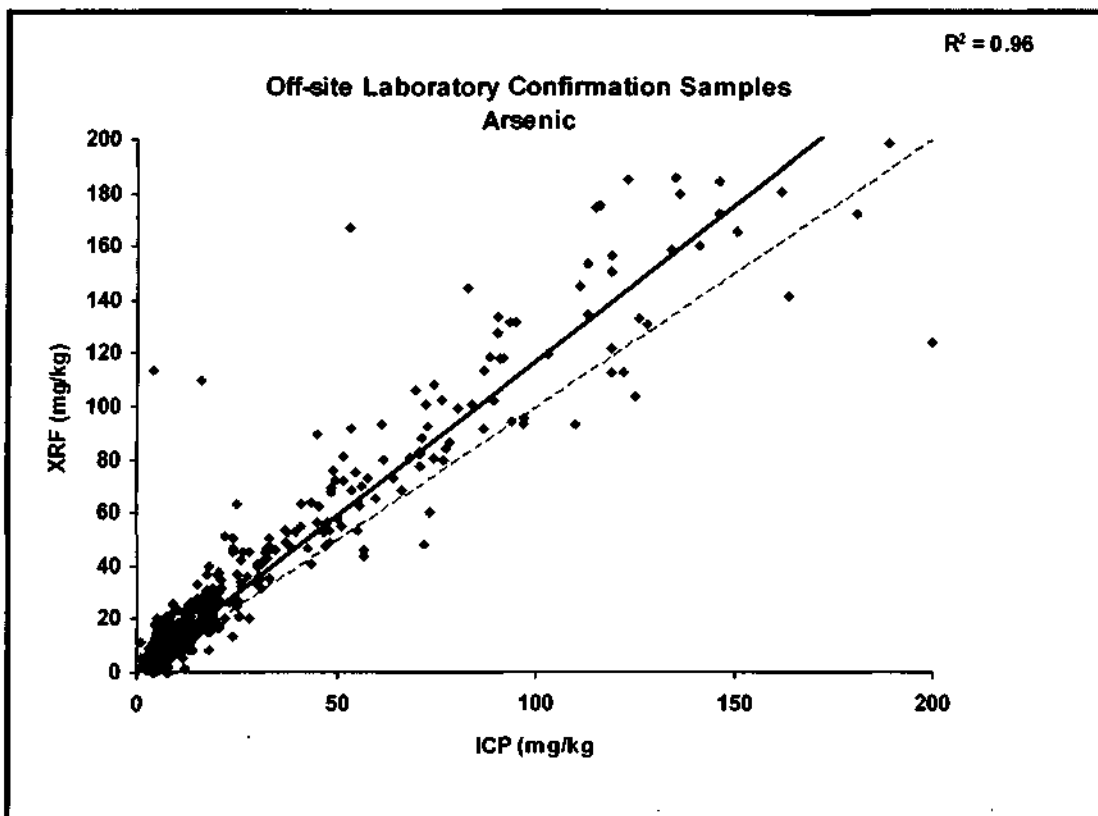
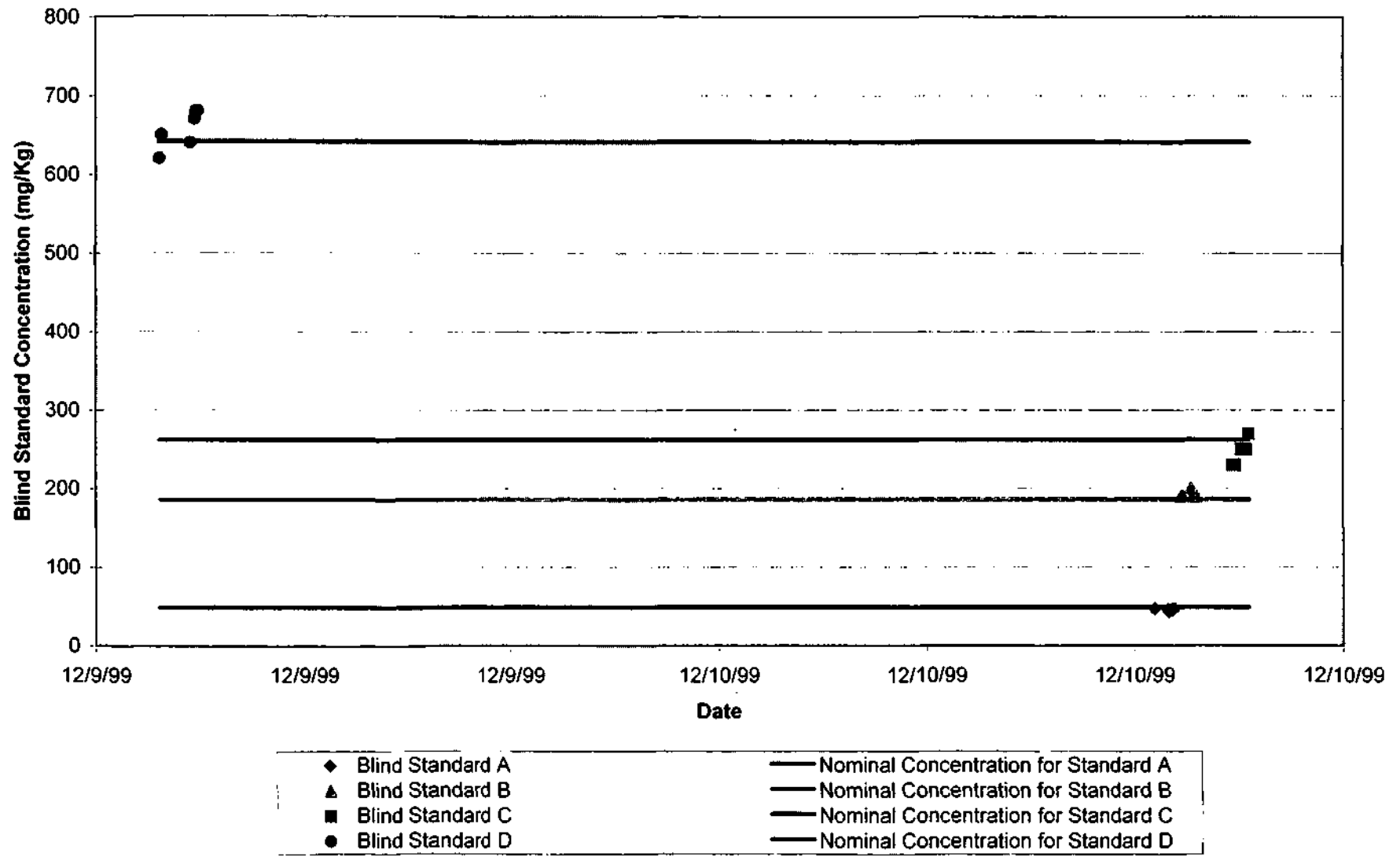


Figure 17



**Figure 18**

**Dust Blind Standard A, B, C, and D - Arsenic**



**Figure 19**

**Dust Blind Standard A, B, C, and D - Lead**

